

Identification of Sources of Lead in Children in a Primary Zinc–Lead Smelter Environment

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We compared high-precision lead isotopic ratios in deciduous teeth and environmental samples to evaluate sources of lead in 10 children from six houses in a primary zinc–lead smelter community at North Lake Macquarie, New South Wales, Australia. Teeth were sectioned to allow identification of lead exposure *in utero* and in early childhood. Blood lead levels in the children ranged from 10 to 42 µg/dL and remained elevated for a number of years. For most children, only a small contribution to tooth lead can be attributed to gasoline and paint sources. In one child with a blood lead concentration of 19.7 µg/dL, paint could account for about 45% of lead in her blood. Comparison of isotopic ratios of tooth lead levels with those from vacuum cleaner dust, dust-fall accumulation, surface wipes, ceiling (attic) dust, and an estimation of the smelter emissions indicates that from approximately 55 to 100% of lead could be derived from the smelter. For a blood sample from another child, > 90% of lead could be derived from the smelter. We found varying amounts of *in utero*-derived lead in the teeth. Despite the contaminated environment and high blood lead concentrations in the children, the levels of lead in the teeth are surprisingly low compared with those measured in children from other lead mining and smelting communities. **Key words:** children, environmental samples, isotopes, lead, smelter, teeth. *Environ Health Perspect* 112:52–60 (2004). doi:10.1289/ehp.6465 available via <http://dx.doi.org/> [Online 25 September 2003]

It is well recognized that in the past, processing of lead–zinc and zinc–lead ores in smelters has resulted in widespread contamination of the environment and has severely affected the health of the community, especially young children. Despite the introduction of emission controls through regulatory guidelines of, for example, lead in air, mining and smelting operations may continue to contaminate the environment and humans (Esterman and Maynard 1998; Hiltz 2003; Hunter Health 2003; Morrison 2003a; Van Alphen 1999).

Legal action has been instigated by community members against lead processing companies. Such was the case in a class action involving some 600 people from Port Pirie, South Australia (location of the largest lead–zinc smelter in the world), and North Lake Macquarie, 120 km north of Sydney, Australia (Gordon 2002; Pasmenco Ltd. 2000a).

Although smelters and/or mines are the obvious point sources in these communities, other lead sources may be present and may contribute to elevated blood lead concentrations, especially in children. For example, on the basis of total lead concentrations, Kimbrough et al. (1995) suggested that lead in paint together with the condition of the house accounted for 12% of blood lead variance in children from a community in which a smelter had closed. Similarly, in the Broken Hill mining community, New South Wales (NSW), Australia, high-precision lead isotopes demonstrated that paint and gasoline were contributors to blood lead in families, although the dominant source was lead derived from the

local ore bodies (Gulson 1996; Gulson et al. 1996a, 1996b).

In response to requests for assistance from members of the North Lake Macquarie community and pediatrician Graham Vimpani, we have undertaken lead isotopic analyses of environmental and biologic samples, collected over a decade, to determine the sources of lead, especially in the teeth of children. In some cases, the parents were concerned that their children may have been exposed *in utero*. In this article we present the results of these investigations, which illustrate the potential of the lead isotopic fingerprinting method in determining lead sources in such communities but, because of funding constraints, are neither systematic nor exhaustive.

Materials and Methods

Setting/history. The community of North Lake Macquarie is located approximately 120 km north of Sydney, NSW, Australia, and consists of three suburbs (Boolaroo, Argenton, Speers Point), with approximately 1,600 households. Three junior schools for pupils 5–12 years old are located in the area.

A primary zinc–lead smelter, Cockle Creek Smelter Pty. Ltd., is located within this community (Figure 1) and is currently operated by Pasmenco Ltd. The smelter began production in 1897, ceased operation in 1922, resumed again in 1961, and closed in September 2003. It produces zinc, lead, and sulfuric acid as major products.

In 1991, an investigation was undertaken by the Hunter Area Public Health Unit.

This consisted of measurements of blood lead in children 1–13 years old in Boolaroo and Argenton, along with measurements of lead in soil, household dust, and paint. The results showed that 6% of children 1–4 years old had blood lead levels > 25 µg/dL and 84% had blood lead levels > 10 µg/dL (Galvin et al. 1993).

There was a significant relationship between the higher blood lead levels and proximity to the smelter, with the exception of slag (glassy smelter residue) infills (Galvin et al. 1993; Unpublished data). In addition, the soil survey (202 samples) reported lead values ≤ 21,460 ppm in First Street, directly opposite the smelter, with decreasing values away from the smelter. Furthermore, there was widespread use of slag as landfill, mainly in recreational areas, with levels ≤ 6,000 ppm Pb as far as 2 km from the smelter and one value ≤ 15,210 ppm Pb, 1 km from the smelter (Galvin et al. 1993). Although the metals in slags are considered to be safely contained within the glassy matrix (e.g., Body et al. 1988), Morrison (2003b) has shown that the fine fractions of the Cockle Creek slags have a very high bioaccessibility.

After disclosure of the results of the Hunter Area Public Health Unit investigations, a Community Consultative Committee was formed that later became known as the Lake Macquarie Environmental Health Coordination and Liaison Committee.

In 1992, decontamination work was undertaken at the Boolaroo school. At the same time, the local government body instituted a

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notation of contaminated land on property certificates within the Hunter Area Health Service sampling program, as a way to protect potential purchasers. This produced anxiety about property values and compounded concerns about health issues. Pasmafinco Ltd. purchased houses from residents who lived in a "buffer zone," which is the eastern section of the first two streets in Boolaroo and closest to the smelter (Figure 1; Lunn and Rothery 2000).

In 1995, a commission of inquiry for environment and planning (CIEP; 1995) was held in response to an application by Pasmafinco Ltd. to upgrade the smelter capacity. An outcome of this inquiry was 63 conditions of consent

relating to environmental controls. These included a program to reduce smelter lead pollution exposure, designed to remediate historical sources of lead and dust and to reduce ongoing ambient lead deposition through plant improvements. The commission of inquiry also recommended the establishment of the North Lake Macquarie Remediation Management Committee, with \$A4.5 million funding from the NSW Environment Protection Authority, NSW Health, and Pasmafinco Ltd.

In 1996, the Environmental Health Centre was established at Boolaroo to undertake individual house remediation, blood lead monitoring, and case management. In 1998, a

2-year strategic plan was developed to include individual house and zonal remediation, blood lead monitoring and case management, greening, education, marketing and health promotion, and monitoring of ambient air quality. The remediation program is directed by the Environmental Health Centre and supervised by the NSW Department of Public Works and Services. The aim of the Environmental Health Centre was to reduce the blood lead levels of children in North Lake Macquarie to the national goal of $< 10 \mu\text{g}/\text{dL}$.

Despite improved emission control by the company and the intense intervention/remediation programs, 37% of 87 children younger than 5 years tested from July 2001 to June 2002 had blood lead levels of $\geq 10 \mu\text{g}/\text{dL}$ (Hunter Health 2003).

Sampling. Variable numbers of samples were taken from six houses (Figure 1). In no house was it possible to obtain all biologic and environmental samples. For example, only two blood samples were taken because, at the beginning of this study, we thought that teeth would provide a more satisfactory history of the lead exposure of the children, given the success of investigations using teeth at Broken Hill (Gulson 1996; Gulson and Wilson 1994). In other houses, most of the environmental samples were obtained except for ceiling (attic) dusts. Sampling protocols generally followed those described by Gulson et al. (1996c).

Subjects and sampling details. A brief history of the children follows. Where possible, teeth were also obtained from siblings.

House 1. This family lived in the house for 14 years, approximately 400 m from the smelter boundary but just outside the buffer zone. The mother had been raised in a nearby suburb, and both female siblings were born in Boolaroo. The younger sibling had a blood lead level of $31 \mu\text{g}/\text{dL}$ at the time of the first visit to the pediatrician, and this remained $> 25 \mu\text{g}/\text{dL}$ for more than 2 years despite efforts to clean up the house and yard. She has progressed poorly at school and has experienced behavioral problems. Her sister is 3 years older and had a blood lead of $24.5 \mu\text{g}/\text{dL}$ at 10 years of age; she also had some learning problems. The older brother had a blood lead of $21 \mu\text{g}/\text{dL}$ at 14 years of age. The blood lead of the mother was $11 \mu\text{g}/\text{dL}$. The children did not play in the "dirt," according to the mother. Sampling of this house was undertaken by the public health unit and showed lead levels in the ceiling (attic) dust of 19,000 ppm, soil levels of 1,100 ppm, and vacuum cleaner dust levels of 1,200 ppm.

House 2. The following information is extracted with permission from the LEAD [Lead Education and Abatement Design group] *Action News* (Mosman 1997), from Gordon (2002), and from assessors' reports

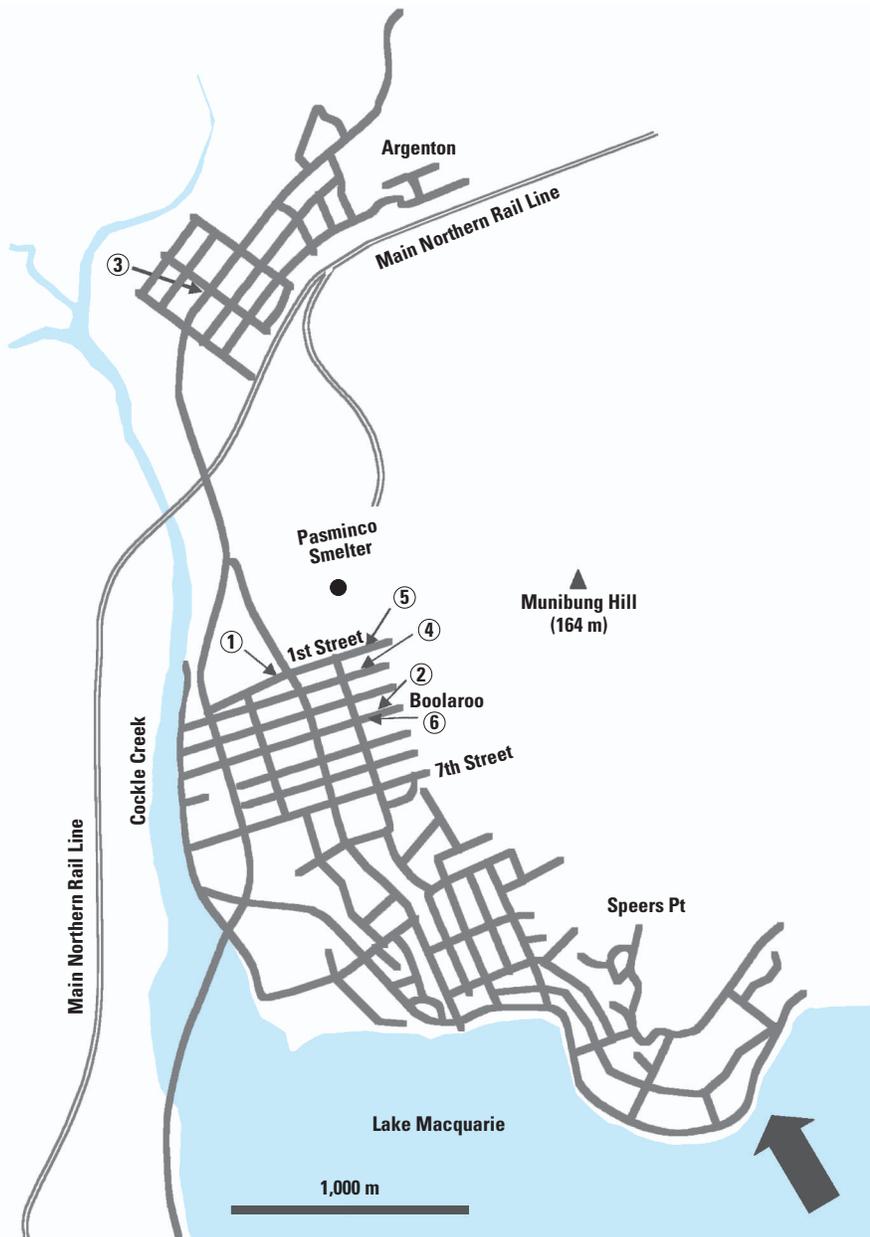


Figure 1. Location of the Cockle Creek smelter in relation to surrounding suburbs and houses sampled (numbered 1–6). House 7 is located approximately 3 km north of the smelter. Arrow points to north.

(Waller G. Unpublished data) supplied by the householder. This house is located approximately 550 m downwind from the smelter boundary but outside the buffer zone. There were three children 15, 9, and 3 years old at the time of sampling in April 1998; the family had moved into the house from a low-lead environment in November 1990. The mother had a blood lead level of 13 $\mu\text{g}/\text{dL}$. The older daughter was 4 years of age when the family moved into the house. In 1995 the youngest girl, at age 3, suffered health problems with stomach pains, loss of appetite, constipation, and high temperature. She also experienced respiratory problems and lost the use of her legs. A bone scan showed possible lead lines, and a follow-up blood test showed a lead level of 34 $\mu\text{g}/\text{dL}$. An environmental assessment of the home by a lead assessor showed the following values: front yard soil, 2,650 ppm Pb; backyard soil, 1,010 ppm; ceiling dust, 15,800 ppm (lead loading of 3,160,000 $\mu\text{g}/\text{m}^2$ or 3.16 g/m^2); the front bedroom window well where the child slept had a lead loading of 12,333 $\mu\text{g}/\text{m}^2$. Paintwork throughout the house was in excellent condition, although comprehensive testing for our investigations showed leaded paint in the lower (older) layers. The house was remediated in the beginning of 1997 through the Environmental Health Centre. However, repeat tests 7 months later by the same assessor showed that the front wall of the house (facing the smelter), which had been completely repainted, had a lead loading of 5,935 $\mu\text{g}/\text{m}^2$, compared with a preremediation level of 2,950 $\mu\text{g}/\text{m}^2$. A 12-month follow-up showed that the remediated ceiling cavity contained dust with 10,240 ppm Pb (lead loading of 13,820 $\mu\text{g}/\text{m}^2$). Samples of ceiling dust were collected in two areas, one from the open area of the ceiling and another in a location not easily accessible which contained deposits up to 2 cm thick of a crusted orange dust. Dust fall accumulation was measured by leaving petri dishes in the ceiling and frequented locations in the house for about 3 months. Dust wipes were also collected in a number of locations, especially the younger child's bedroom. For comparison, a petri dish was placed in the lounge room of the adjacent neighbor's house. Teeth were available from the two girls.

House 3. This house is approximately 1 km north of the smelter in the direction of the main summer prevailing winds (Galvin et al. Unpublished data). The front wall of the house is located approximately 20 m from a main thoroughfare. The mother, pregnant at the time, moved into the house in September 1995 from a newly built house in a low-lead environment, and started to renovate. At 26 weeks of pregnancy, she went into labor, and the baby lived for 12 hr. She again undertook renovations during her second pregnancy, the following year. The baby had blood lead

levels of 22 $\mu\text{g}/\text{dL}$ at 10 months and 14 $\mu\text{g}/\text{dL}$ at 15 months. The front yard was well grassed but the soil, assessed by the independent assessor for the Environmental Health Centre, had a concentration of 82,000 ppm Pb, probably due to the presence of dispersed slag particles, clearly present in the samples. Additional soil samples were collected at the front and rear yards and under the house. Exterior and interior paint was in reasonable condition. Wipes were collected from the front walls of the house, an outer verandah wall, and window sills of two front windows. The baby's room had been renovated, but the remainder of the house was in a state of disrepair, with gaps between ceilings and walls in a number of rooms and carpets in poor condition. Petri dishes were placed in the dining room, lounge room, and child's bedroom. At the time of sampling in July 1998, the family was not living in the house.

House 4. The house is located approximately 500 m from the smelter stack. The mother was raised in a suburb approximately 2 km east of the smelter that was subject to smelter emissions during her childhood, especially sulfur dioxide. The family moved into the Boolaroo house in about 1976. The parents carried out a lot of renovations when the children were very young. The mother's blood lead was never measured. The boy spent time in the back yard moving surface dirt with his toy trucks. His blood lead was 14 and 15 $\mu\text{g}/\text{dL}$ when he was 9 and 10 years old, respectively. He has major learning and behavioral difficulties and was assessed at one stage to be 5 years behind in school. His younger sister had blood lead levels of 6–8 $\mu\text{g}/\text{dL}$. She played mainly indoors and experienced no learning or behavioral problems. No environmental or blood samples were collected from this house, although the public health unit analyzed a sample of ceiling dust and found it to contain up to 9,000 ppm Pb.

House 5. This family moved into their house, located across the street from the smelter boundary, in 1990. Their former abode had low lead exposure, but both parents had grown up in Boolaroo. The mother had lived in Boolaroo since she was 10 years old; her blood lead was 17 $\mu\text{g}/\text{dL}$. The female child was 18 months old when the family moved to Boolaroo and had a maximum blood lead of 38 $\mu\text{g}/\text{dL}$. The male child was born in 1990, and his maximum blood lead was 42 $\mu\text{g}/\text{dL}$. Both children played on the front verandah facing the smelter, and this was cleaned every day by the mother. Both children had major learning difficulties and behavioral problems and required speech therapy. Environmental samples measured by a lead assessor showed that soil in both front and rear yards contained more than 4,000 ppm Pb, ceiling dust contained about 2,760 ppm, vacuum cleaner

dust 2,650 ppm, and the top of a ceiling fan 10,670 ppm. No environmental or blood samples were available for lead isotope analysis.

House 6. This family lives within approximately 500 m of the smelter boundary and in the direction of prevailing winds. The blood lead of both parents was < 10 $\mu\text{g}/\text{dL}$. The girl had a blood lead of 28 $\mu\text{g}/\text{dL}$ at age 4 but did not appear to have any learning difficulties. The boy had a blood lead of 38 $\mu\text{g}/\text{dL}$ at 18 months old and was diagnosed as autistic at a later stage. He has learning and speech difficulties. The parents renovated their house, including removing a false ceiling, while the mother was pregnant with the son. To minimize contribution from leaded paint, the husband replaced the exterior of the house with new cladding. The house underwent remediation in 1999. About 18 months after remediation, the ceiling dust contained 15,800 ppm Pb in the 250 μm fraction (Morrison A. Personal communication). No blood samples were available for analysis for lead isotopes.

House 7. This house is located approximately 3 km north of the smelter. The family moved into the house in 1992 from a low-lead environment. The two girls, 6 and 7 years old at time of exposure, have been diagnosed with autism. One girl with a blood lead level of 20 $\mu\text{g}/\text{dL}$ has pica, and the high blood lead was attributed to slag present in the soil. Inspection of several soil samples, especially those from where the girls played most and ate dirt, showed the presence of lead paint flakes and a very complex mixture of other particles. Lead paint samples from the house, lead paint grains from the soil, and blood samples were analyzed.

Analytic methods. Environmental and blood samples were prepared following the methods described by Gulson et al. (1996c).

Gulson and Wilson (1994) and Gulson (1996) demonstrated the use of the lead isotope technique, combined with the well-established histology of teeth, in evaluating *in utero* and early childhood lead exposure from slices of deciduous teeth. In this approach, analysis of the enamel provides evidence of *in utero* exposure. Analysis of dentin provides evidence of exposure during the early childhood years, when hand-to-mouth activity is usually an important contributor to lead body burden, and potentially up to the time of tooth exfoliation.

In children exposed to lead sources from mining, paint, or gasoline in communities such as the Broken Hill lead mining community, it has been shown that the source of lead from the incisal sections was different from the source of lead in the cervical sections of deciduous teeth, reflecting the change in lead from exposure *in utero* to that in early childhood (Gulson 1996; Gulson and Wilson 1994).

The deciduous teeth were crowns only because the roots had been resorbed. Wherever possible, central and/or lateral incisors were analyzed to allow comparison with other studies. The crowns of both upper and lower incisors were cut transversely into slices 1–2 mm thick from the incisal and cervical areas using a diamond-impregnated stainless steel disk. The incisal section consisted of enamel and varying amounts of coronal dentin. Because enamel and coronal dentin are formed before the crawling stage of most children (Lunt and Law 1974; Orban 1953), they provide an indicator for lead exposure *in utero* and in earliest childhood. For determinations on enamel samples, as much attached dentin as possible was removed, and vice versa. The lower central portion of the incisal section was reamed to ensure removal of any later-formed circumpulpal dentin.

As much as possible of the thin veneer of enamel was cut from the cervical section

using a dental bur, leaving mainly dentin. The pulpal canal in the cervical section was usually resorbed to varying degrees, but approximately 2 mm of the pulpal canal and dentin was reamed out, to ensure minimal contribution from secondary (circumpulpal) dentin. The dentin in the cervical section provides an integrated exposure to lead from the time of eruption of the tooth until exfoliation (Purchase and Fergusson 1986; Rabinowitz et al. 1989, 1991).

After a rigorous washing procedure described by Gulson (1996), a ^{202}Pb “spike” solution of known isotopic composition and lead concentration was added to the tooth sample to obtain the concentration of lead and isotopic composition of the unknown sample in the one analysis. The methods of analysis are described in detail by Gulson and Wilson (1994) and Gulson (1996).

Because of the relatively low lead concentrations observed in the teeth of some of these

children who had high blood lead concentrations over periods extending apparently to several years, we were concerned that our rigorous tooth “cleaning” procedure with hydrogen peroxide may have removed much of the lead. To evaluate this, a central incisor was sliced sagittally (lengthwise) and then in half, and the enamel/dentin sections prepared as described above. The tooth sections were then subjected to a 3-hr peroxide leach, rinsed, dried, reweighed, and subjected to a further leach for 21–24 hr. The tooth section was then cleaned with our normal methods and analyzed. Leachates were spiked with ^{202}Pb and analyzed.

Results

Because of the limited number of samples, it was not possible to rigorously analyze the data using statistical methods, a condition also found by Gwiazda and Smith (2000) and Manton et al. (2000).

Table 1. Isotopic and lead concentration data for samples from house 1.

Sample type	$^{208}\text{Pb}:^{206}\text{Pb}$	$^{207}\text{Pb}:^{206}\text{Pb}$	$^{206}\text{Pb}:^{204}\text{Pb}$	$^{207}\text{Pb}:^{204}\text{Pb}$	Pb (ppm)	Date collected
Soil, front yard (–100 μm) ^a						
1	2.1516	0.9002	17.25	15.52	3,090	29 Apr 1993
2	2.1560	0.9043	17.15	15.51	4,130	29 Apr 1993
3	2.1555	0.9022	17.22	15.53	4,700	29 Apr 1993
Soil, backyard (–100 μm) ^a						
7	2.1465	0.8965	17.30	15.51	740	29 Apr 1993
8	2.1468	0.8952	17.38	15.56	3,340	29 Apr 1993
Paint, wall ^b						
1	2.1994	0.9366	16.54	15.49	3,820	29 Apr 1993
2	2.1654	0.9140	16.94	15.49	110	29 Apr 1993
Vacuum cleaner, house dust (–100 μm) ^c						
1	2.1435	0.8913	17.62	15.70	3,320	29 Apr 1993
2	2.1385	0.8899	17.50	15.57	3,280	29 Apr 1993
3	2.1385	0.8902	17.51	15.59	3,610	29 Apr 1993
Vacuum cleaner, bedroom	2.1276	0.8790	17.76	15.61	6,960	29 Apr 1993

^aSample numbers indicate separate soil sample sites. ^bSample numbers indicate difference locations on the house. ^cSample numbers indicate subsamples from a single bag.

Table 2. Isotopic and lead concentration data for samples from houses 2 and 6.

Sample type	$^{208}\text{Pb}:^{206}\text{Pb}$	$^{207}\text{Pb}:^{206}\text{Pb}$	$^{206}\text{Pb}:^{204}\text{Pb}$	$^{207}\text{Pb}:^{204}\text{Pb}$	Pb (ppm)	Date collected
Petri, dust (adjacent neighbor)	2.1511	0.8966	17.38	15.58	1,240 $\mu\text{g}/\text{m}^2/30$ days	17 Jul 1998
Petri, dust	2.1383	0.8893	17.47	15.54	4,780 $\mu\text{g}/\text{m}^2/30$ days	17 Jul 1998
Wipe, window sill	2.1481	0.8958	17.35	15.55	510 $\mu\text{g}/\text{m}^2/30$ days	08 Apr 1998
Wipe, ceiling dust	2.1824	0.9204	16.90	15.55	480 $\mu\text{g}/\text{m}^2/30$ days	08 Apr 1998
Ceiling, dust crust	2.2153	0.9514	16.19	15.40	780 ppm	08 Apr 1998
Ceiling, dust (neighbor)	2.1438	0.8927	17.43	15.56	15,800 ppm	19 Feb 2003

Table 3. Isotopic and lead concentration data for samples from house 3.

Sample type	$^{208}\text{Pb}:^{206}\text{Pb}$	$^{207}\text{Pb}:^{206}\text{Pb}$	$^{206}\text{Pb}:^{204}\text{Pb}$	$^{207}\text{Pb}:^{204}\text{Pb}$	Pb	Date collected
Blood from baby	2.1478	0.8976	17.32	15.55	13.6 $\mu\text{g}/\text{dL}$	11 Aug 1998
Paint, front house ^a	2.2102	0.9453	16.35	15.46	NM	17 Jul 1998
Paint, outer sheds attached to house ^a	2.2196	0.9557	16.10	15.39	NM	17 Jul 1998
Soil, backyard	2.1659	0.9123	16.95	15.47	490 ppm	17 Jul 1998
Soil, front yard	2.1471	0.8968	17.29	15.51	530 ppm	17 Jul 1998
Soil, under house	2.1369	0.8831	17.70	15.63	1270 ppm	17 Jul 1998
Wipe, front veranda wall	2.1320	0.8855	17.55	15.54	980 $\mu\text{g}/\text{m}^2$	17 Jul 1998
Wipe, front wall	2.1308	0.8845	17.56	15.53	500 $\mu\text{g}/\text{m}^2$	17 Jul 1998
Wipe, front window sill	2.1285	0.8833	17.58	15.52	57,000 $\mu\text{g}/\text{m}^2$	17 Jul 1998
Petri, dust from child's room	2.1340	0.8844	17.63	15.59	505 $\mu\text{g}/\text{m}^2/30$ days	22 Sep 1998
Petri, dust from dining room	2.1436	0.8914	17.50	15.60	1,143 $\mu\text{g}/\text{m}^2/30$ days	22 Sep 1998
Petri, dust from front room	2.1358	0.8894	17.53	15.59	41,014 $\mu\text{g}/\text{m}^2/30$ days	22 Sep 1998

NM, not measured.

^aRapid reaction with lead test.

The results are presented in Tables 1–5 and graphically as an isotope ratio plot (Figure 2) of the type conventionally used in isotope geochemistry and less commonly in environmental health investigations (e.g., Gulson et al. 1996c; Rabinowitz and Wetherill 1972).

The superiority of lead isotope analyses over elemental lead analyses lies in their ability to assign proportions to the contributing lead sources. The success of this process is contingent on there being sufficient difference in the lead isotopic ratios in the source materials and the ability to detect this analytically. The larger the difference in isotopic composition between the sources, the more definitive are estimations of source apportionments. Australia is unique because the isotopic compositions of its main sources of lead come

from deposits that are geologically old (formed about 1,700–1,800 million years ago). The ²⁰⁶Pb:²⁰⁴Pb ratio in these deposits is 16.0–16.1; smaller and geologically younger deposits from the same continent formed about 400–500 million years ago have a ²⁰⁶Pb:²⁰⁴Pb ratio of about 18.1–18.3 (Gulson 1986). The isotopic contrast between these two deposit groups is about 100 times our experimental error for blood samples. In contrast, Manton et al. (2000) stated that in the United States, where the isotopic differences between sources are generally much smaller than in Australia, the lead isotope method provides satisfactory discrimination in only one in five cases.

In simple cases where there is two-component mixing of lead (i.e., two main sources or

end members), the isotopic data define linear trends on plots of isotopic ratios as shown in Figure 2. For any point *X* lying on that linear trend, it is possible to estimate the proportion of each of the end members (em) or sources by the following relationship:

$$\text{Proportion of sample } X (\%) = \frac{{}^{206}\text{Pb}:{}^{204}\text{Pb}_x - {}^{206}\text{Pb}:{}^{204}\text{Pb}_{\text{em1}}}{{}^{206}\text{Pb}:{}^{204}\text{Pb}_{\text{em2}} - {}^{206}\text{Pb}:{}^{204}\text{Pb}_{\text{em1}}} \times 100$$

An example of the calculation is given for the slag in the following section.

Possible sources of lead and their lead isotopic fingerprints. Smelter emissions/slag. An indirect measure of the isotopic composition of smelter emissions can be derived from the analysis of slag samples. However, slag samples represent only a limited time interval because their composition varies in consonance with the feedstock for the smelter.

A slag sample from July 1998 has an isotopic ratio of 17.78 (Table 4, Figure 2), which shows that the lead in the feedstock at that

Table 4. Isotopic data for slag samples collected.

Sample type	²⁰⁸ Pb: ²⁰⁶ Pb	²⁰⁷ Pb: ²⁰⁶ Pb	²⁰⁶ Pb: ²⁰⁴ Pb	²⁰⁷ Pb: ²⁰⁴ Pb	Date collected
Slag 1	2.1268	0.8774	17.78	15.60	11 Aug 1998
Slag 2	2.1288	0.8782	17.78	15.61	11 Aug 1998

Table 5. Isotopic and lead concentration for teeth samples.

House	Sex	Max PbB (age)	Tooth type	Section	²⁰⁸ Pb: ²⁰⁶ Pb	²⁰⁷ Pb: ²⁰⁶ Pb	²⁰⁶ Pb: ²⁰⁴ Pb	²⁰⁷ Pb: ²⁰⁴ Pb	Pb (ppm)	Date collected	
1	F		Lower canine	E	2.1674	0.9136	16.96	15.50	2.2	23 Aug 1993	
				D	2.1613	0.9104	17.02	15.49	13.3	23 Aug 1993	
				Cent inc	E	2.1341	0.8866	17.56	15.57	0.8	23 Aug 1993
				D	2.1361	0.8915	17.43	15.54	1.4	23 Aug 1993	
1	F	31 (5)	Left upper inc	E	2.1509	0.9008	17.19	15.48	7.4	09 Feb 1993	
				D	2.1500	0.8993	17.24	15.50	19.9	09 Feb 1993	
2	F		2nd molar	?E	2.1696	0.9176	16.89	15.50	0.5	05 Feb 1998	
				?D	2.1714	0.9196	16.82	15.47	1.8	05 Feb 1998	
2	F	34 (4)	Lower cent inc	E	2.1678	0.9133	16.96	15.49	3.1	22 Jun 1998	
				D	2.1691	0.9132	16.93	15.46	6.1	22 Jun 1998	
			Upper L inc	E	2.1670	0.9133	16.95	15.48	2.2	21 Feb 2000	
				D	2.1568	0.9069	17.06	15.47	2.5	21 Feb 2000	
			Upper cent inc	E	2.1585	0.9081	17.06	15.49	4.3	31 Jan 2000	
				D	2.1616	0.9103	17.00	15.48	5.7	31 Jan 2000	
4	M	15 (10)	Upper canine	E	2.1537	0.9025	17.19	15.52	0.6	09 Dec 1999	
				D	2.1381	0.8906	17.43	15.52	5.5	09 Dec 1999	
				Root	2.1351	0.8889	17.48	15.54	5.6	09 Dec 1999	
4	F	8 (10)	Upper canine	E	2.1542	0.9027	17.19	15.52	0.6	09 Dec 1999	
				D	2.1387	0.8906	17.45	15.54	3.4	09 Dec 1999	
5	F	38 (?)	Upper cent inc	E	2.1558	0.9040	17.18	15.53	1.5	09 Dec 1999	
				D	2.1536	0.9031	17.18	15.52	2.2	09 Dec 1999	
			Upper L inc	E-1	2.1525	0.9026	17.16	15.49	2.5	21 Feb 2000	
				Tooth cut into quarters	3-hr leach	2.1172	0.8736	17.80	15.55	2.7 ng	21 Feb 2000
			21-hr leach	E-2	2.1500	0.9013	17.19	15.50	3.0	21 Feb 2000	
				3-hr leach	2.1254	0.8751	17.70	15.48	2.5 ng	21 Feb 2000	
			21-hr leach	D-1	2.1423	0.8903	17.49	15.57	2.2 ng	21 Feb 2000	
				D-1	2.1452	0.8973	17.25	15.48	4.5	21 Feb 2000	
			3-hr leach	D-1	2.1231	0.8779	17.68	15.52	3 ng	21 Feb 2000	
				24-hr leach	2.1297	0.8837	17.65	15.60	2.6 ng	21 Feb 2000	
			D-2	D-2	2.1455	0.8972	17.27	15.49	6.0	21 Feb 2000	
				3-hr leach	—	—	—	—	2.8 ng	21 Feb 2000	
			21-hr leach	2.1209	0.8759	17.81	15.60	1.6 ng	21 Feb 2000		
			5	M	42 (?)	Upper cent inc	E	2.1455	0.8972	17.28	15.50
D	2.1476	0.8986					17.25	15.50	4.6	09 Dec 1999	
6	F	28 (4)	Lower L inc	E	2.1449	0.8960	17.32	15.52	0.8	05 Feb 1998	
				D	2.1499	0.8998	17.22	15.49	1.5	05 Feb 1998	
6	M	38 (1.5)	Lower LL inc	D	2.1464	0.8972	17.29	15.51	6.7	05 Feb 1998	
				Lower RL inc	E	2.1388	0.8911	17.41	15.51	2.6	05 Feb 1998
					D	2.1467	0.8983	17.25	15.49	5.3	05 Feb 1998

Abbreviations: —, data not sufficient quality; cent, central; D, dominantly dentin; E, dominantly enamel; F, female; inc, incisor; L, lateral; LL, left lateral; M, male; Max PbB (age), maximum blood lead concentration in micrograms per deciliter and age at which it is measured; RL, right lateral.

time was derived mainly from geologically young Australian mineral deposits, such as those found at Woodlawn and Elura (Cobar district) in NSW and/or at Tasmanian mines such as Rosebery, Que River, and Hellyer (em2). If, as is likely, the geologically old component in the feedstock was from the Broken Hill and/or Mt. Isa mines (em1), then the proportion of geologically young lead in the slag sample can be estimated, using the above relationship, at about 90%. Before the exploitation of the Woodlawn and Elura mines in the mid-1970s, the source of feedstock to the smelter was from mines in the Broken Hill area (Firkin 1980). Hence, in the early days of the smelter, the $^{206}\text{Pb}:^{204}\text{Pb}$ ratio of the feedstock, slag, and emissions would have been closer to 16.0, the $^{206}\text{Pb}:^{204}\text{Pb}$ value for Broken Hill mines.

Since 1969, the range of materials processed by the Cockle Creek smelter has been widened to include material from the Broken Hill and Cobar districts in western NSW and from Tasmania (Firkin 1980). Given the figures for raw material mass balance provided in Firkin (1980) and our unpublished lead isotope data, it is possible to estimate the isotopic fingerprint for the feedstock for the smelter. This calculation, probably representative of the 1970s material and later (because smelter feedstocks are varied as little as possible), gives a $^{206}\text{Pb}:^{204}\text{Pb}$ ratio of 17.4, a value lying in the range measured for dust, as described below. A calculation employing similar methodology for emissions from the lead-zinc smelter in the early 1990s at Risdon, Tasmania (also operated by Pasmenco Ltd.), using information on the feedstock provided by the company, has

previously given a consistent result for the isotopic composition of the feedstock and that of dust in air in a suburb adjacent to the smelter (Gulson et al. 1996c).

Leaded gasoline. The isotopic fingerprint for gasoline can be obtained in two ways, from a direct measure of the lead in gasoline and from air monitoring. From a comparison of lead isotopic measurements in gasoline and high-volume air filters, Chiaradia et al. (1997) showed that more than 90% of the lead in Sydney air was derived from gasoline and that high-volume air filters provide valid information on the gasoline source. Because the gasoline supplies for Newcastle are predominantly from Sydney, it is valid to use the analyses of gasoline and air filters (Chiardadia et al. 1997).

Mean values of the isotope ratios in Sydney high-volume air filters for 1988–1998 show an overall increase in the $^{206}\text{Pb}:^{204}\text{Pb}$ ratio over time (Figure 2). The line of best fit through the mean data has a slope that is considerably lower than the slope for the soils and teeth.

Paint. Because any house in Australia built before 1970 could contain lead paint (Environment Australia 1999) and many of the houses, especially those closest to the smelter, are more than 50 years old, we expected that they would contain leaded paint. A concern was that the paint history may be very complex because houses from older parts of Sydney could contain up to 13 different layers of leaded paint (Gulson et al. 1995).

Because such complexity might mask any potential use of lead isotopic measurements in paint at Boolaroo, a systematic study of the paints in one house (house 2) was undertaken. The isotopic composition of lead-rich

paint samples both from house 2 and from the other houses has a relatively restricted range (Figure 2), with $^{206}\text{Pb}:^{204}\text{Pb}$ ratios clustering around a value of about 16.0, although one sample has a value up to 16.5. The isotopic ratios in most paint samples are characteristic of the lead derived from the Broken Hill mines.

Diet. Although we undertook no dietary investigation, we have been measuring dietary intake in various Australian communities for more than a decade, usually employing a 6-day duplicate diet approach. We have found that diet is generally a minor contributor to blood lead in the Australian context (Gulson et al. 1996c, 1997, 1999) and expect that the same would apply in a community such as North Lake Macquarie.

Environmental samples. Soils. There is a large variation in the isotopic composition for the soils, with the $^{206}\text{Pb}:^{204}\text{Pb}$ ratio ranging from 16.9 to 17.5 (Figure 2). This variation probably reflects changes in the feedstock to the smelter over time from when the feedstock had a lower $^{206}\text{Pb}:^{204}\text{Pb}$ ratio, although in some cases there may be a small contribution from lead paint and/or gasoline. A similar pattern of lower $^{206}\text{Pb}:^{204}\text{Pb}$ ratios in soils compared with house dust and house air was observed in the area surrounding the Port Pirie smelter (Gulson et al. 1996c). We concluded that this pattern once again reflected the early use of predominantly Broken Hill feedstock (Body et al. 1988) with a $^{206}\text{Pb}:^{204}\text{Pb}$ ratio of 16.0.

Dust. Dust wiped from hard surfaces, dust fall accumulation from petri dishes, and vacuum cleaner dust represent current sources of smelter lead.

Deposition rates can be extremely high. For example, wipes from the exterior of house 3 contained 57,000 $\mu\text{g Pb}/\text{m}^2$ despite the cleaning of the exterior of this house by the owner approximately 3 weeks before the sampling. Likewise, monthly dust accumulation from the interior of houses located within 800 m of the smelter ranges up to 4,000 $\mu\text{g Pb}/\text{m}^2/30$ days (Table 3). These high values may be compared with considerably lower values measured in Sydney over a decade, where the arithmetic mean value of 298 measurements was $83 \pm 92 \mu\text{g Pb}/\text{m}^2/30$ days and the range was 6–1,046 $\mu\text{g Pb}/\text{m}^2/30$ days.

Isotopic ratios in the dust accumulations lie within a relatively restricted range from 17.35 to 17.63 and are considered to represent mainly the emissions from the smelter during the 1990s. The sample from house 6 contained 15,800 ppm Pb (Table 2) and was deposited, postremediation, over approximately 18 months from 2000 to 2001. The $^{206}\text{Pb}:^{204}\text{Pb}$ ratio of 17.44 lies within the range for the 1990s data. The dust data, however, have slightly lower $^{206}\text{Pb}:^{204}\text{Pb}$ values than

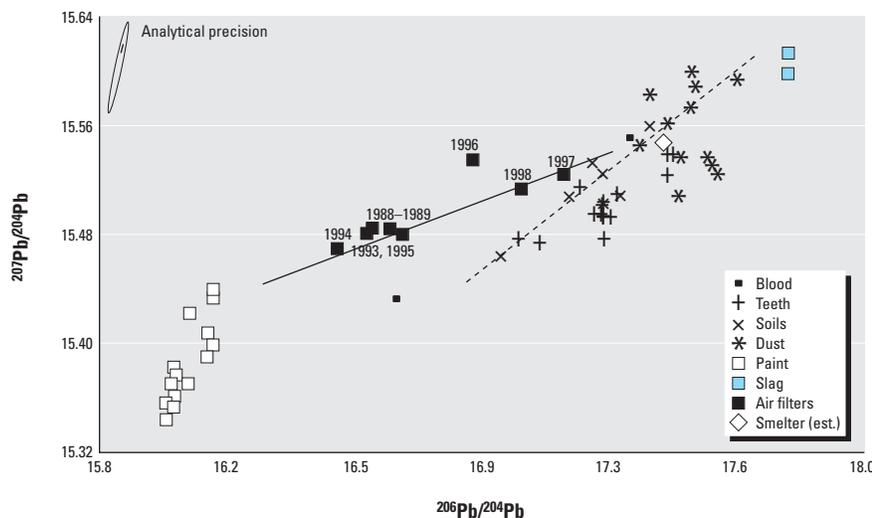


Figure 2. Isotope ratio plot for the various environmental and biologic samples, using data from Tables 1–5 and paint samples from house 7. The estimate of the smelter emissions is for the period covering the 1970s, using information on the feedstock from Firkin (1980). The air filters are from Sydney, containing > 90% lead from gasoline (Chiaradia et al. 1997). Dust wipes include dust from vacuum cleaner, surface wipes, ceiling (attic), and dust-fall accumulation over 3 months using petri dishes. The dashed and solid lines are the best fits through the data for teeth and air filters, respectively. The 95% error ellipse for replicate analyses of samples and standards is shown in the upper left corner.

those for the late 1990s slag. These values are identical with the ratio of 17.4 obtained from the feedstock figures listed in Firkin (1980) and are probably representative of the mid- to late 1970s.

Careful sampling within the ceiling cavities can also provide important information on feedstock change over time. For example, in house 2, inadequate removal of ceiling dust meant that, in difficult-to-reach areas, "historical" samples could be obtained. This solidified orange dust had a $^{206}\text{Pb}:^{204}\text{Pb}$ ratio of 16.19 (Table 2), reflecting the early feedstock for the smelter from the Broken Hill mines, whereas the "current" ceiling dust from this house had a $^{206}\text{Pb}:^{204}\text{Pb}$ ratio of 17.47 (Table 2). During "explosions" at the smelter, orange clouds of dust have been described as entering houses (Mosman 1997).

Teeth. Lead isotopic composition and lead concentration data for deciduous teeth from several children are listed in Table 5. The data for dentin, considered to represent the lead exposure during early childhood, are plotted in Figure 2.

Leaching experiments showed that the unexpectedly low lead concentrations of tooth lead in most children were not the result of leaching with peroxide during the cleaning process. The amount of lead extracted during the peroxide leach ranged from 1.6 to 3 ng Pb, < 3% of the total amount of lead present in the sample of tooth analyzed. Isotopic ratios of most leachates also showed that negligible amounts of lead are extracted from the tooth during the wash; the higher $^{206}\text{Pb}:^{204}\text{Pb}$ ratios in the leachates probably derive from the peroxide, whose $^{206}\text{Pb}:^{204}\text{Pb}$ ratio is 18.1.

The patterns for lead concentrations observed in deciduous teeth from most of these subjects are much the same as we observed in earlier studies in mining environments (Gulson 1996; Gulson and Wilson 1994). That is, the lead concentrations in the enamel, derived predominantly from the mother, can be an order of magnitude less than in dentin, which reflects exposure during early childhood. For example, in siblings from house 4, the lead concentrations were 0.55 and 0.51 ppm in the enamel and 5.5 and 3.4 ppm in the dentin for the boy and girl, respectively. The differences in lead concentrations in the dentin are consistent with the maximum blood lead concentrations of 15 $\mu\text{g}/\text{dL}$ for the boy and 10 $\mu\text{g}/\text{dL}$ for the girl. In other sibling pairs from house 5 and house 6, the lead concentrations in dentin are only about twice those of the enamel despite their very high maximum blood lead concentrations of 28–42 $\mu\text{g}/\text{dL}$.

Isotopic compositions in enamel and dentin show no consistent relationships, in contrast to those from Broken Hill, probably because of the limited number of samples and any changes in feedstock to the smelter.

Blood. We analyzed only two blood samples isotopically. In one case (Table 3), the isotopic results are the same as those in a sample of soil from the front yard (in which slag grains were present) but lower than the current smelter products, evidenced in the wipes and petri dust. We estimate that > 90% of the lead in the blood was derived from current smelter activities.

In the other case (house 7), where lead paint flakes were common in the soil and the child had pica, about 45% of lead in her blood was estimated to be derived from paint. The identification of other lead sources is complicated by the complex history of the land and its past use, perhaps for disposal of industrial waste (Davis J. Personal communication; Morrison A. Personal communication). A contribution to lead burden from emissions from the smelter is unlikely, because Huang and Gulson (2002) have shown that beyond about 3–4 km from the smelter, in both northerly and southerly directions, levels of lead in soil approximates those in background soils.

Discussion

Sources of lead. This limited study illustrates the complexities involved in trying to establish sources of lead in children in a community centered around a lead industry. Use of lead concentrations alone would not have provided answers to indicated sources, given the patterns of lead paint in the houses, former use of lead in gasoline, and the dominant presence of the smelter.

The isotopic measurements demonstrate that the contribution of lead from paint to tooth and blood lead levels is minor, except for the child from house 7. Likewise, the contribution to body burden from gasoline lead is small because of the decreasing use of leaded gasoline, the relatively low traffic density in this area, location of the houses relative to any moderately heavy-traffic thoroughfare, and the difference in slope of the data for the high-volume air filters representing gasoline lead (Figure 2).

Thus, the only credible remaining source is from the smelting activities that have been carried out for more than 100 years.

Pathways of lead. Besides direct inhalation of the stack emissions, the main pathway of lead to the children is through ingestion of soil and dust.

Soil. High concentrations of lead in the soil can result from stack emissions (Pasmenco Ltd. 2000b), fugitive emissions (Court 2001), and the use of slag as fill (Galvin et al. Unpublished data; Morrison 2003b).

The range of lead isotopic ratios shown in Figure 2 can arise from several factors, including changes in the feedstock, such as varying mixtures of geologically old lead with low $^{206}\text{Pb}:^{204}\text{Pb}$ ratios of about 16.0 and geologically

young ore with $^{206}\text{Pb}:^{204}\text{Pb}$ ratios of about 18.1; a contribution from lead paint with low $^{206}\text{Pb}:^{204}\text{Pb}$ values; and past use of gasoline. As argued above, gasoline is a minor contributor to lead burden in North Lake Macquarie. The lead paint can be a significant contributor, and in the case of two households (houses 1 and 7), lead paint flakes in soil were identified and analyzed.

Dust. Dust is now recognized as the major contributor to blood lead. In urban areas, dust is derived mainly from lead paint (Clark et al. 1985; Jacobs et al. 2002; Lanphear et al. 1999, 2000, 2002) and from former use of leaded gasoline (Mielke et al. 1997, 1999). In communities with lead activities, the most obvious source of the dust was from mining or smelting processes (Bornschein et al. 1985; Gulson et al. 1994, 1996a, 1996b; Hiltz 2003; Landrigan et al. 1975; von Lindern et al. 2003a, 2003b).

In the present study, we used the dust from a variety of media, including vacuum cleaner dust, surface wipes, dust fall accumulation in petri dishes over 3-month periods, and ceiling dust. These media provide information for dust accumulation over periods of days in the case of, say, window ledge wipes (house 2; Tables 2 and 3) to over a period of about 18 months for ceiling dust accumulation (Table 2).

Considering the 5 years during which the dust samples were collected and the diversity of dust types, the isotopic results showed limited variation. If it is conceded that the overwhelming majority of lead in the dust derives from smelting activities, the relatively small isotopic range in the dust samples would imply that the feedstock to the smelter was relatively constant over this time interval.

Teeth. Most of the isotopic data for tooth dentin exhibit a shift to the left of the dust data and overlap the trend for the soils (Figure 2). This could be interpreted to mean that the soils were a more significant contributor to lead in tooth dentin and the dust or that another lesser source of lead was present, such as gasoline and/or lead paint. Gasoline lead is considered a minor contributor to that in the environment, teeth, and blood in this area. However, for at least one set of siblings, from house 4, the isotopic composition in the dentin is the same as the dust from the neighboring houses.

Using the estimations discussed above for the isotopic composition of the smelter emissions based on dusts and feedstock materials, approximately 55–100% of the lead in teeth could derive from the smelter. This estimation assumes that paint is the other end member for the calculation, rather than gasoline.

From this and other studies, we suggest that if the enamel lead in deciduous teeth is < 1 ppm and possibly closer to 0.5 ppm, these low levels reflect a minimal *in utero* exposure of the child. For many children from North Lake

Macquarie, it would appear that significant amounts of lead have been transferred to the child. Low levels in enamel were observed only in siblings from house 4, and the similarity in isotopic composition of enamel and dentin for both siblings indicates exposure to a similar source of lead. Despite tooth differences that can affect lead concentrations based on whole tooth analyses (Lockeretz 1975; Purchase and Fergusson 1986; Tvinnereim et al. 2000), the levels of lead in enamel were generally > 2 ppm for the North Lake Macquarie children. In subjects where the ratio of lead in dentin:enamel is 2:1, there has been potentially a higher amount transferred from the mother, and this may mask the amounts added to dentin during early childhood.

Except for siblings from house 1—which may reflect a higher level of exposure (Table 1) from the mid- to late 1980s and early 1990s than that of the other families—the lead concentrations in the teeth from the North Lake Macquarie children were unexpectedly low given the widespread contamination of the environment. For comparison, children with higher exposure in the Broken Hill lead-zinc mining community have a mean enamel lead concentration of 3.1 ppm (range, 1.0–8.9) and a mean dentin lead concentration of 12.9 ppm (range, 3.0–31.5; Gulson 1996). Children living in the vicinity of a United Kingdom smelter had whole crown levels of 12.3 ppm (1.4–340) compared with control levels of 4.7 ppm Pb; these represent a time of exposure in the mid- to late 1970s (Delves et al. 1982). The mean whole tooth lead was 6.4 ppm [95% confidence interval (CI), 4.2–10.0] for children living in the area around a Czech smelter, compared with background levels of 1.5 ppm Pb (Cikrt et al. 1997). This study covered children exposed in the late 1980s and early 1990s whose mean blood lead level for the most contaminated area around the smelter was 15.4 µg/dL (95% CI, 7.2–33.1 µg/dL). The mean tooth lead in 262 incisors from 262 children living around the Port Pirie lead-zinc smelter in South Australia was 8.6 ± 1.9 µg/g, and the correlation of tooth and blood lead at age 7 years was 0.78 ($p < 0.001$; McMichael et al. 1994).

The source of the enamel lead could be the increased amounts released from maternal bones during the last half of pregnancy (Franklin et al. 1997; Gulson et al. 1998) and/or from ambient lead. Unfortunately, the exposure of the mothers is rarely reported, although in one case (house 5) the mother's blood lead was 17 µg/dL, and in two other cases (houses 1 and 2) the mother's blood lead was ≥ 11 µg/dL.

There are several limitations to this study, the major deficiency being the limited number of blood samples. A better designed study

would require, besides the blood samples, direct measurements of smelter emissions from appropriately placed dust accumulation devices and high-volume air filter samplers, more wipes including hand wipes of the children, and better information on feedstock materials. Nevertheless, the results demonstrate that lead isotopes remain the most powerful tool for evaluating sources of lead in the environment and in humans, and despite continuing use, there are a limited number of practitioners using this technique.

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